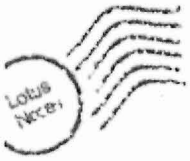

Carol Lynes/R2/USEPA/US
10/25/2006 03:31 PM

To jchristian@kelso.caslab.com
cc Mark Reiss/R2/USEPA/US@EPA
bcc
Subject Oct. 17, 2006 Letter Re: Chemical Data Assessment - Sampling and Testing of Material Proposed for Dredging from San Juan Harbor Section 103, Puerto Rico

History: This message has been replied to.

Vice President Christian,

As you requested, I am acknowledging receipt of your October 17, 2006 letter, regarding the EPA's evaluation of the analytical data submitted for the above referenced project. Both Mark Reiss and I reviewed your letter and appreciate your honesty regarding the events that occurred.

Project planning is a critical quality assurance component to the success of a project. EPA strongly encourages pre-project planning meetings to avoid any confusion in the program and project specific requirements when preparing a quality assurance project plan for submission to EPA.

Both Mark and I are willing to take part in the pre-project planning phase of future dredging projects that you may provide analytical services for in Region 2.

Respectfully,

Carol L. Lynes
EPA Region 2
Division of Environmental Science and Assessment
732-321-6760
732-321-6616 (fax)

1317 South 13th Avenue P.O. Box 479 Kelso, Washington 98626 (360) 577-7222 ph (360) 636-1068 fax

October 17, 2006

Carol L. Lynes, Environmental Scientist
U.S.E.P.A. – Region II
2890 Woodbridge Ave.
Building 209, MS-220
Edison, NJ 08837-3679

Re: Chemical Data Assessment – Sampling and Testing of Material Proposed for Dredging from the San Juan Harbor Section 103, Puerto Rico

Dear Ms. Lynes:

This letter is in response to your document dated June 1, 2006 concerning the data assessment for elutriate and tissue analyses performed by Columbia Analytical Services, Inc. (CAS) at our Kelso (WA) laboratory in support of the San Juan Harbor dredge project.

Note that I apologize for the tardiness of this response, which is partially due to outstanding circumstances surrounding this project, but also due to less than ideal internal communication at our Kelso laboratory as related to the management of the project. In addition, we conducted a thorough investigation into the various items listed in your report.

Please note that we take your evaluation extremely serious and hope to resolve all items for future analytical chemistry performed in support of Region II projects. The biggest single cause to the deviations from the Quality Assurance Plan (QAP) for this work was the lack of preliminary review of the requirements on our part. The QAP and the Regional Testing Manual (RTM) do not contain information foreign to CAS. We are accustomed to performing projects that meet specifications as per those required for this work.

As mentioned, we failed to perform a thorough review of the QAP and consequently omitted components to the analytical chemistry work deemed mandatory by Region II. On behalf of our organization, I apologize for the oversight.

The following categories represent the major items or topics referenced in your report. I have added discussion of each item to help clarify the cause and to define our protocol in the future.

Method Detection Limits (MRL) and Method Reporting Limits (MDL):
Several comments were noted regarding MRLs and MDLs.

- MDL studies are performed annually at CAS for all analytes in each matrix as applicable. For tissue, organic extractables are performed in analyte-free tissue (i.e. commercially available tissue that we test to assure native concentrations are not high enough to skew MDL studies). For metals, actual tissue cannot be used due to the elevated concentration of numerous target analytes.

  For seawater, CAS has not historically used 'open ocean seawater' for MDL studies, but can do that for extractable organics when required. For metals, that could be done as well, but most elements would not be spiked due to ambient concentrations significantly greater than our MDLs. Formulating seawater matrices from synthetic mixes cannot be done for metals due to trace contaminants in the salts. Refer to the section on SRMs for additional information related to performance in seawater.

- The MDL study submitted for metals in tissue was expressed on an equivalent dry basis because metals data is generally evaluated in tissue on that basis. When requested to report values on both a dry weight and wet weight basis, the MDLs are calculated based on the moisture content of the sample. When we submit an MDL study that is expressed on wet weight basis, the values are adjusted to reflect 80% moisture, which is a level that represents a typical value for tissue. In hindsight, we should have submitted the MDLs expressed on both dry and wet basis.

- The relationship between the MRL and MDL varies to some degree. For organics, the MRL is derived from the low point in the calibration curve. For metals, the MRL is generally ~3-5 times greater than the associated MDL, although some exceptions exist. In no case does CAS report values less than the experimentally determined MDL.

- The fluctuation in MDL and MRL from sample to sample is a function of using different aliquot sizes for the determination. Although the aliquots are generally very close, they are not normally exactly equal. The reporting software adjusts the MDL and MRL based on the initial mass or volume. In addition, the MRL and MDL are adjusted when dilutions are performed.

Standard Reference Material (SRM) and Laboratory Control Sample (LCS):
Generally speaking, the handling of SRMs by CAS for this project was unacceptable. The responsibility of our Project Chemists is to review the requirements for the analytical work and communicate any requirements outside the scope of our default QA/QC protocol or operating procedures to the laboratory staff. We have default QA/QC requirements that meet our internal protocol, as well as our state and federal regulatory

protocol. However, in many instances (including this project) we are required to operate under additional or alternative protocol. Obviously, this was not done, particularly as it related to the analysis of SRMs.

Please note the specific comments related to the individual matrices and analytical fractions.

- **Metals in Seawater:** Our laboratory typically analyzes an NRC seawater SRM when requested. Any future work done under the RTM will include a seawater SRM.
- **Metals in Tissue:** NRC tissue SRMs are analyzed with every batch. However, the reporting of the results was not consistent with the requirement for this project. This is an issue that can be resolved for future work under the RTM.
- **Organics in Seawater:** Since SRMs are not available for aqueous matrices, this item will require discussion for future work under the RTM. A synthetic seawater mix might be appropriate or the use of open ocean seawater. Again, preliminary discussion and planning will be the key to satisfying this item.
- **Organics in Tissue:** The analysis of an SRM for organics (Pesticides, PCB Congeners, and PAHs in this case) presents a little more complex situation. The biggest problem with this project was that our laboratory did not analyze a tissue SRM for all fractions. Results from an analysis close to the time these samples were analyzed were provided after the fact. The approval date represented the date the results were sent, not the date extracted and analyzed. Regardless, providing SRM results unassociated with the batch was of little to no value for the samples on this project.

  The other issue related to the analysis of SRMs in tissue consists of choosing an SRM with certified values derived from the same or very similar procedures specified for the project. This is important if we are required to meet the recovery information specified in the RTM. Typical NIST or similar SRMs have certified values derived from procedures outside the scope of EPA Methods. Values are assigned via a number of techniques that might include more aggressive solvent extraction, isotope dilution corrections, normalization for matrix spike recovery, normalization for surrogate recovery, or instrumental methods outside the scope of the EPA Methods. We are currently identifying applicable SRMs that can be analyzed and evaluated as per the RTM.

Matrix Spikes:
Two issues were of concern relative to matrix spikes. For Organochlorine Pestcides, 2,4-DDD, 2,4-DDE, and 2,4-DDT were not spiked. These compounds are not on the standard default list of compounds normally analyzed, so they were not spiked. However, our laboratory often is requested to include them and we have systems in place to accommodate that. As with the majority of the other issues associated with this project,

the source of this problem was related to internal communication from the project chemist to the laboratory staff. Future work done under the RTM will include spiking of all compounds.

The other item mentioned was metals. The matrix spike performed for the aqueous samples was done on a seawater sample that was not part of this project (i.e. Batch QC). Although the matrix was appropriate to represent performance for the samples, the requirement to run the spike on one of the samples from this project was not met. Again, the source of the problem was incomplete review of the QAP.

Data Reporting – Identification of Extraction and Analysis Batches:
The comments concerning the lack of clear batch identifications were in reference to the metals work. Samples are assigned batch numbers and each group of twenty samples or less is tracked through the laboratory with an associated set of quality control samples. However, we can understand how the tracking can be confusing to a data reviewer when greater than twenty samples are processed, as was the case with the metals work. This is easily remedied by limiting the number of samples processed per Sample Delivery Group (SDG), which is the normal CAS protocol. Unfortunately, this project was not set up correctly by our Project Chemist.

As I mentioned, we take these matters seriously and accept all responsibility for not meeting your expectations on this project. We have taken corrective action including re-training of staff on standard operating procedures for project management. Future work will be handled according to the project specifications, and at the very least, any exceptions or proposed modifications will be discussed during the initial planning stages of the project.

I would appreciate a brief comment acknowledging receipt of this letter and would welcome further discussion. In addition, we are willing to send laboratory representation to your office to help facilitate our ability to meet your expectations in the future. I can be reached at jchristian@kelso.caslab.com or (360) 501-3316.

Sincerely,

Columbia Analytical Services, Inc.

Jeff Christian
Vice President/Kelso Laboratory Director

Cc: Mark Reiss – U.S. EPA Region II/New York, NY